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RESEARCH AND DEVELOPMENT PROGRAM FOR ROCKET-BORNE
CRYOSORPTION PUMP

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FINAL REPORT

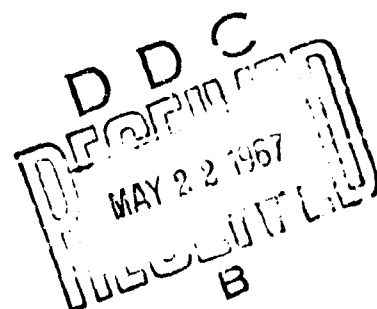Period Covered: 15 May 1965 through 15 July 1966

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ABSTRACT

A cryosorption pump, intended for use with a flight mass
spectrometer, has been constructed and laboratory tested. The
pumping surfaces were formed by first brazing metallized
pellets of Zeolon (a synthetic Zeolite) to copper trays, after
which the overlying braze was machined away, exposing bare sor-
bent. The advantages of this process are that the completed
pump is free of volatile or corrosive residues, can be baked at
temperatures approaching 300°C, and has good thermal contact
between sorbent and heat sink.

The pump was laboratory tested at 77°K, the intended operat-
ing temperature for flight, by admitting controlled bursts of air,
nitrogen, and argon. The shape and intensity of these bursts
simulated those to be expected in flight. It was found that pump
will maintain pressures within the operating limits of the spec-
trometer, and that the speed and total gas capacity of the pump
are superior to those of previously flown cryosorption pumps,
despite the fact that the sorbent areas of the latter pumps sub-
stantially exceed the area of this pump.

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SECTION I

INTRODUCTION

The following report describes the fabrication and testing of a completed cryosorption pump, to be supplied to the Air Force Cambridge Research Laboratory by The NRC Equipment Corporation, under the terms of Contract No. AF19(628)-5171. The pump has been designed to fit inside an existing AFCRL mass spectrometer housing, for use in upper atmospheric soundings. The exterior wall of the AFCRL housing contains a small aperture, to be opened at a predetermined altitude; the ionization chamber of the mass spectrometer is located directly behind this aperture. Throughout the sounding interval, the pump will be required to maintain the gas pressure within the housing at a low enough value to permit operation of the mass spectrometer, and te ensure as well that the proportions of argon and nitrogen within the ionization chamber are representative of those immediately outside the aperture. Laboratory tests of the pump demonstrate that in these regards its performance should far surpass that of cryosorption pumps previously flow by AFCRL.

A novel process has been employed in the construction of this pump, viz., the brazing of metallized sorbent pellets to copper trays, followed by the removal of sufficient material to expose a bare sorbent surface. The process, on which patent application has been filed, has the advantages of providing good thermal and mechanical bonding between the individual sorbent pellets and the heat sink, and of permitting the pump to be baked at temperatures approaching the softening point of the braze. By comparison, the process of epoxy bonding of sorbent to heat sink is inferior, in that it leaves organic residues and can also result in partial clogging of the sorbent. An epoxy bonded cryosorption pump was formerly used with this flight spectrometer; its performance is described in the final report of Contract No. AF19(604)-8510. An alternative process, the mechanical bonding of sorbent to heat sink by wire screens, is inferior in that it provides relatively poor thermal contact, and may not retain the sorbent under extreme vibration.

To test the performance of this pump, a two chamber vacuum system was constructed, the chambers being coupled by an orifice of the same dimensions as that in the intended flight housing.

The pump was contained in one chamber, together with partial and
total pressure gauges. The pressure in the other chamber was
rapidly varied in such a manner as to simulate the pressure
external to an ascending rocket. The test gases employed were
air, nitrogen, and argon. An analysis was made of the expected
performance of the pump, based on a simple mathematical model,
and the results of this analysis are fairly well supported
by the experimental data. The model should be generally appli-
cable in the design of similar systems, and is decribed in a paper
entitled, "Transient Response of a Cryosorption Pump" which will
be presented at the XIII Annual Symposium of the American Vacuum
Society.

1. Fabrication of Cryosorption Plates

The fabrication process to be described has the fundamental
aim of first providing a clean, mechanically strong, metallic
coating for sorbent pellets, and of then brazing these pellets to
metal trays which serve both as heat sinks and structural members
in the completed pump. The various steps in this process have
been chosen after extensive testing of combinations of materials,
to ensure good wetting of the metallic surfaces, and to further
ensure that no corrosive or volatile residue remains in the com-
pleted pump.

a. Coating Sorbent Pellets

The sorbent consisted of 1/8" diameter pellets of activated
synthetic Zeolite*. These were metalized with a silver coating
about .002" thick. Previous tests had indicated that a coating
thickness of at least .001" would be needed for satisfactory
brazing.

The silver coated pellets were etched for one hour in concen-
trated HCℓ (Assay 37.6% conc.) at a temperature near 100°C. The
pellets were then flushed in running tap water for several hours,
following which they were flushed in deionized water for one
hour. Testing with Hydrion paper indicated a final pH of about
4.5, the same value being obtained for deionized water alone.

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* Zeolon H, a product of the Norton Company, Worcester, Massachusetts.

Figure 1

Cryosorbent panel.

TRAY RIM

SORBENT

1-1/4"

1-3/8"

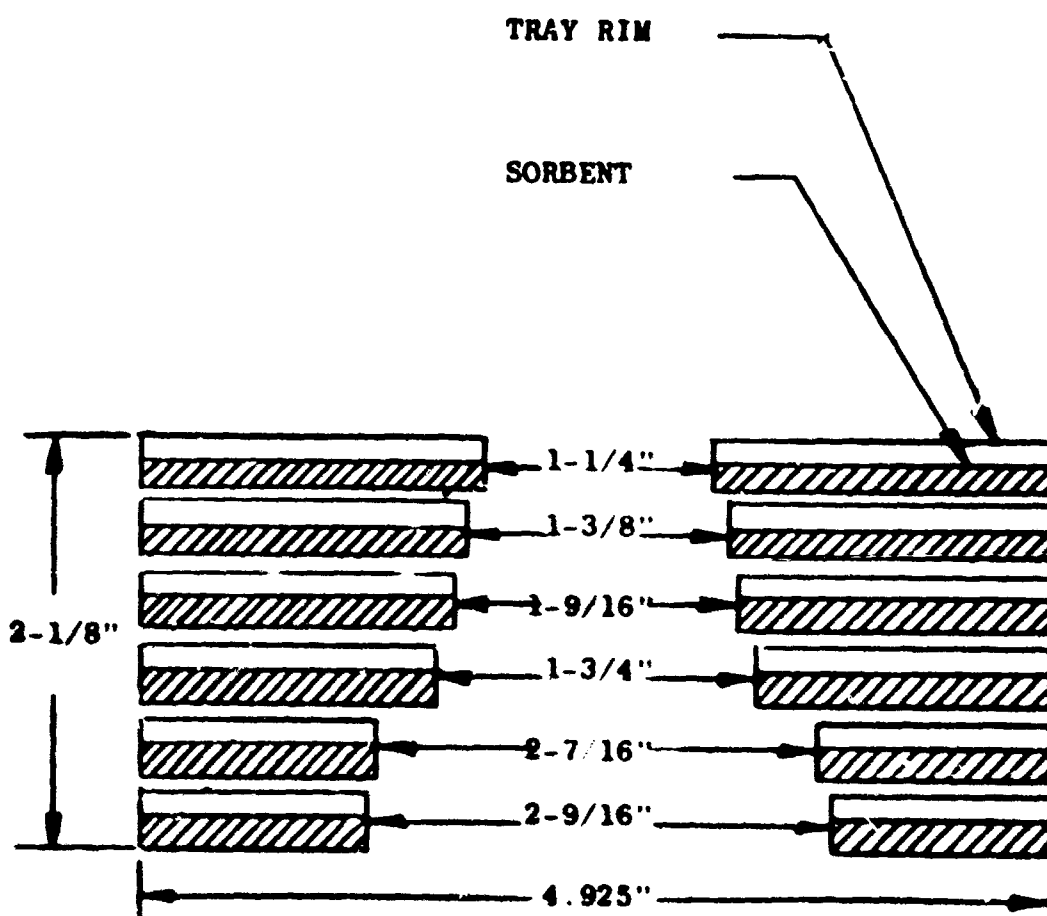1-9/16"

1-3/4"

2-7/16"

2-9/16"

2-1/8"

4.925"

Figure 2

Dimensions of cryosorption pump.

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The clean pellets were then culled to remove any with incomplete coatings; these have a brownish rather than silvery appearance. Finally, the satisfactory pellets were heated in vacuum to drive off adsorbed water. It took several days to completely dry the pellets, the final pressure being about 10^{-6} Torr and the final temperature being about 200°C.

b. **Brazing Pellets to Plates**

The silver-coated pellets were then brazed with pure lead to copper trays. These trays are 1/16" thick, 4.925" in diameter, and have a 1/4" rim to contain the molten lead. The following brazing procedure was employed.

The trays and lead sheets were burnished, washed in acetone, air dried and srayed with Handy Harmon TEC flux.

Three lead sheets, each 5" in diameter and 1/64" thick were placed in each tray, and then heated in air for about 10 minutes at 660°F. After cooling, the trays were burnished, acetone rinsed and air dried. By appearance, the lead had thoroughly wetted the copper.

A densely packed single layer of silvered pellets was then placed in each tray, sprayed with Handy Harmon TEC flux, and covered with two lead sheets 5" in diameter and 1/64" thick. The trays were again heated in air to 660°F for about 20 minutes. Immediately after removing the trays from the furnace, the pellets were pressed into the still molten lead with an aluminum disc. After cooling, the tray was boiled in tap water, rinsed in alcohol, and dried. By appearance, the lead had thoroughly wetted the pellets.

c. **Machining of Cryosorption Plates**

Sufficient material was removed from the surface of the composite trays to expose a bare sorbent surface. The procedure was to chuck the trays in a high-speed lathe, and to then take light, slow cuts across the surface with a blunt, carbide tipped tool bit. A hole was cut in the center of each tray, so that when stacked one above the other, the trays enclose a conical volume which will accomodate the mass spectrometer in the AFCRL flight housing. The uppermost of the six trays comprising the pump is shown in Fig. 1, and the dimensions of the trays are given in Fig. 2. Three 2-56 holes have been tapped radially into the edge of each tray, so that the trays can be anchored to the cylindrical shell during final assembly, this has not yet been done as it may be desired to first inspect the trays.

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SECTION II

TEST APPARATUS

Figure 3 shows the vacuum system used in testing the response of the cryosorption pump to pressure bursts which simulate those to be experienced in atmospheric soundings. The vacuum system has three main sections, which are shown framed by heavy lines in Fig. 3. The uppermost section regulates the pressure bursts, the right-hand section contains the main vacuum pumps, and the left-hand section contains the cryosorption pump and various gauges.

The pressure regulating section consists of a test charge chamber (A), a toggle valve (B), an expansion chamber (C), an orifice (D) leading to the main vacuum system (this is a 1/8" diameter hole in a .010' plate, the plate being clamped between the flanges shown in Fig. 3), an Alphatron* gauge (E) which monitors the pressure burst, and a by-pass valve (F) which is closed during tests.

The main vacuum pump section is entirely conventional, consisting of a cold trap (G), a 6" diffusion pump (H), and a fore-pump (I).

The cryosorption pump section consists of the pump housing in chamber (J), a pinhole orifice (K) leading to the pressure regulating section (this orifice is a .015" hole in a .005" plate, the plate being clamped between the flanges shown in Fig. 3), a total-pressure ionization gauge (L), a mass spectrometer ionization tube (M) and electron multiplier (N), and a bakeable by-pass valve (O) which is closed during tests. Figure 4 is a closer view of the cryosorption pump section alone. Figure 5 is a top view of chamber (J) removed from the system, showing the copper radiation shield which surrounds the cryosorption pump. This shield is bolted to the floor of the chamber (J); during testing, the chamber is immersed in liquid nitrogen to a depth of at least 8". Figure 1 shows the cryosorption pump; the copper straps on the outer wall of the pump casing are bolted to the floor of the chamber (J).

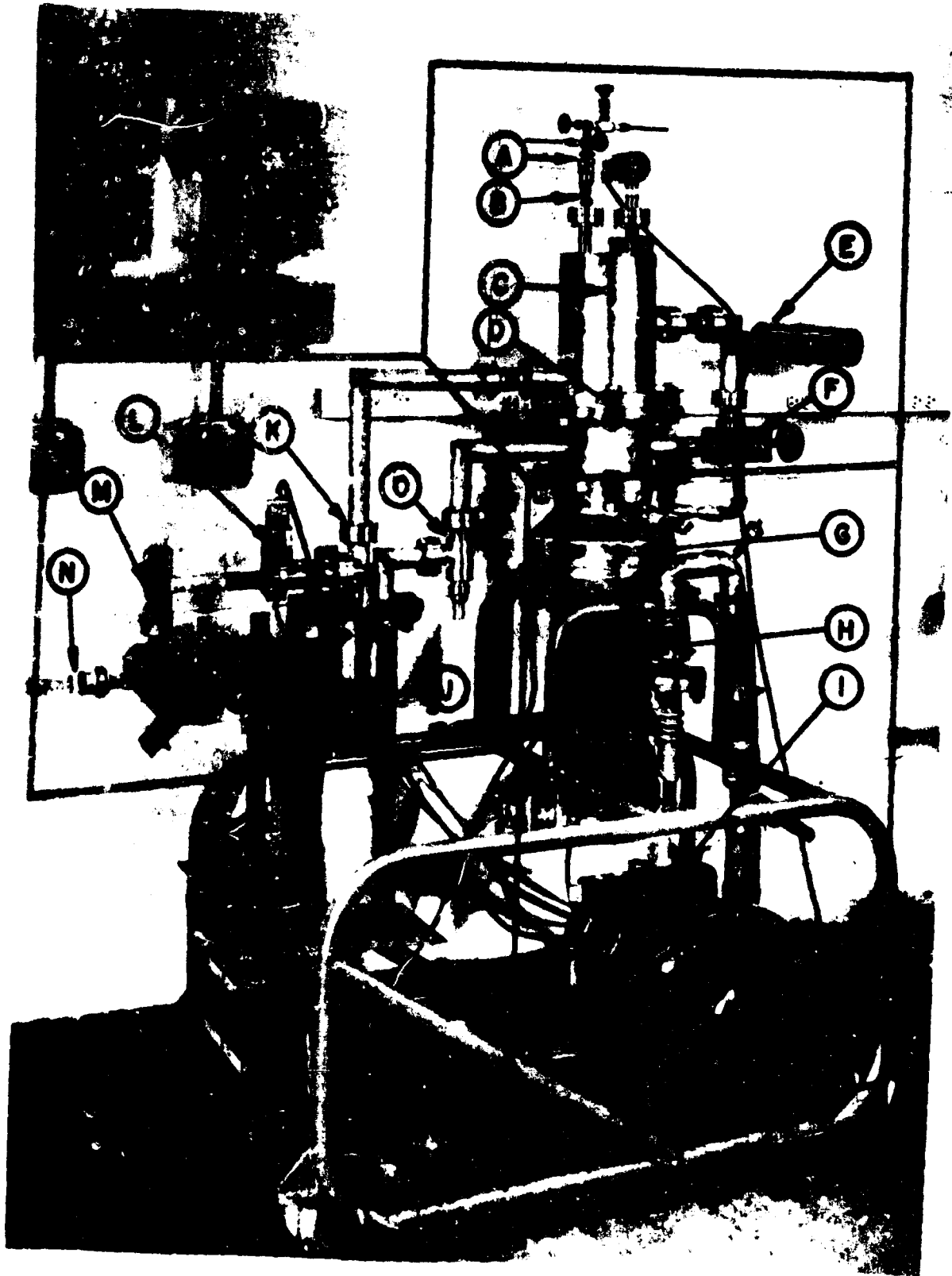* Registered trademark of National Research Corporation.
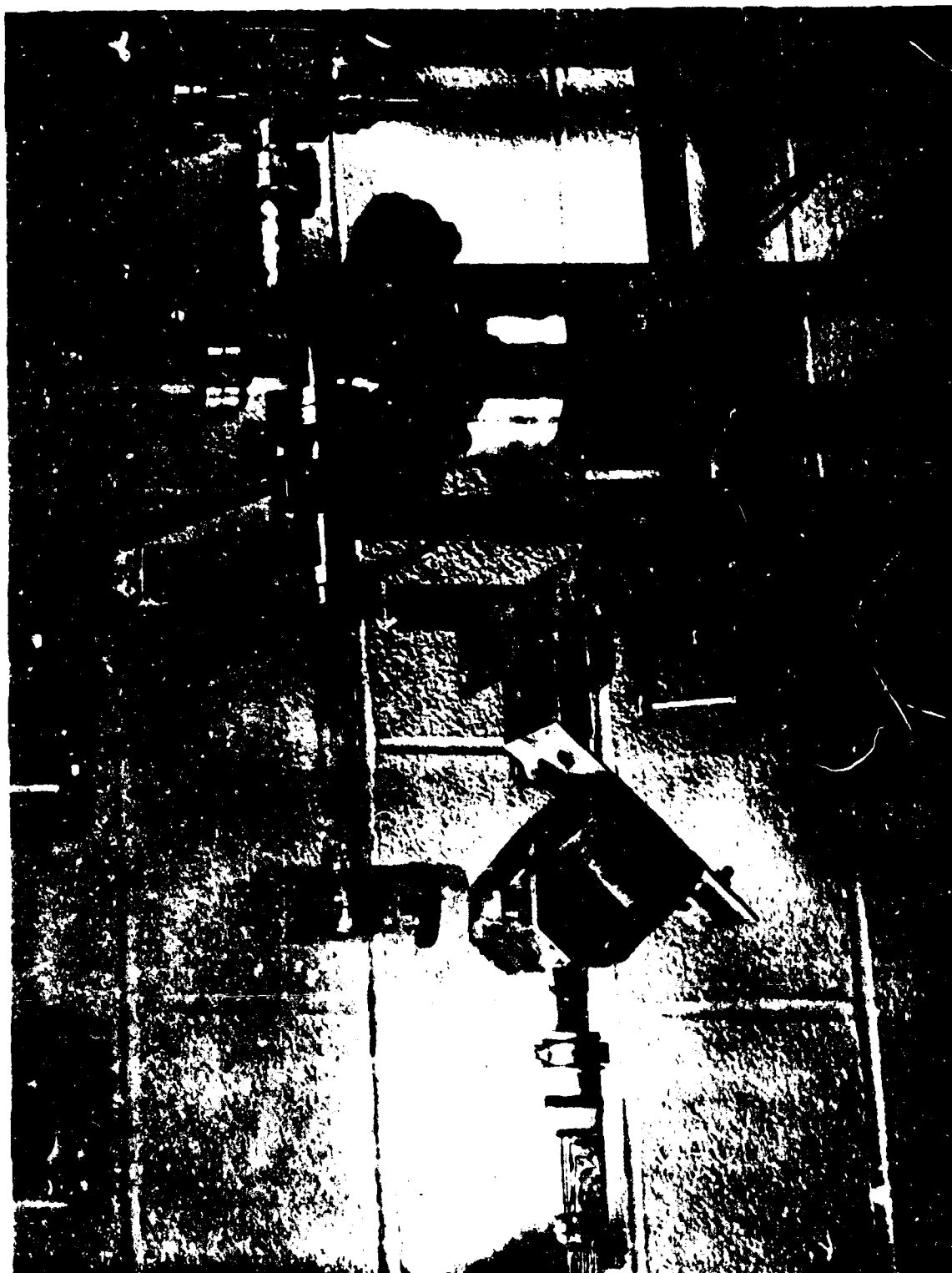
Figure 3

Vacuum test system.

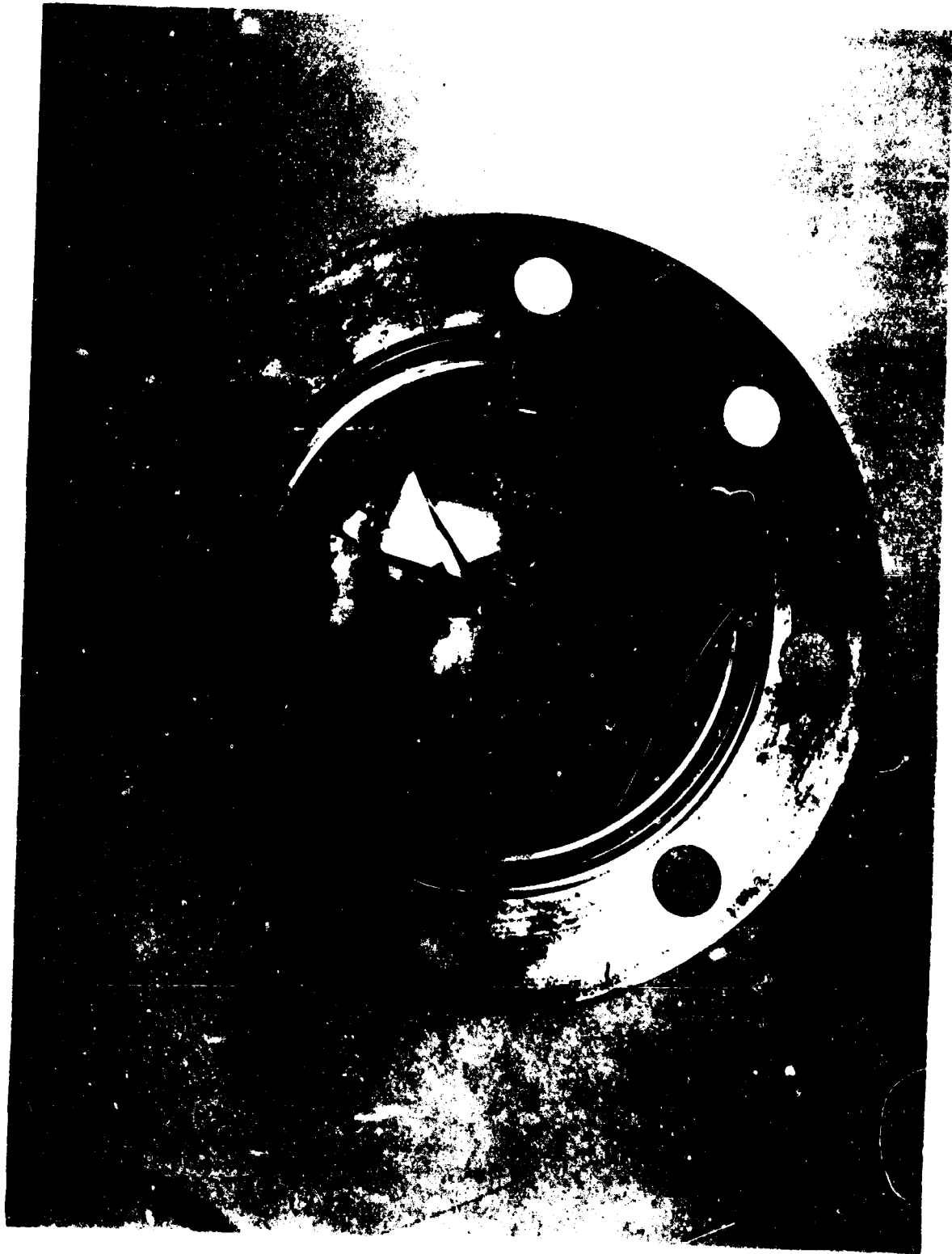Figure 4

Cryosorption pump section of test system.

Figure 5
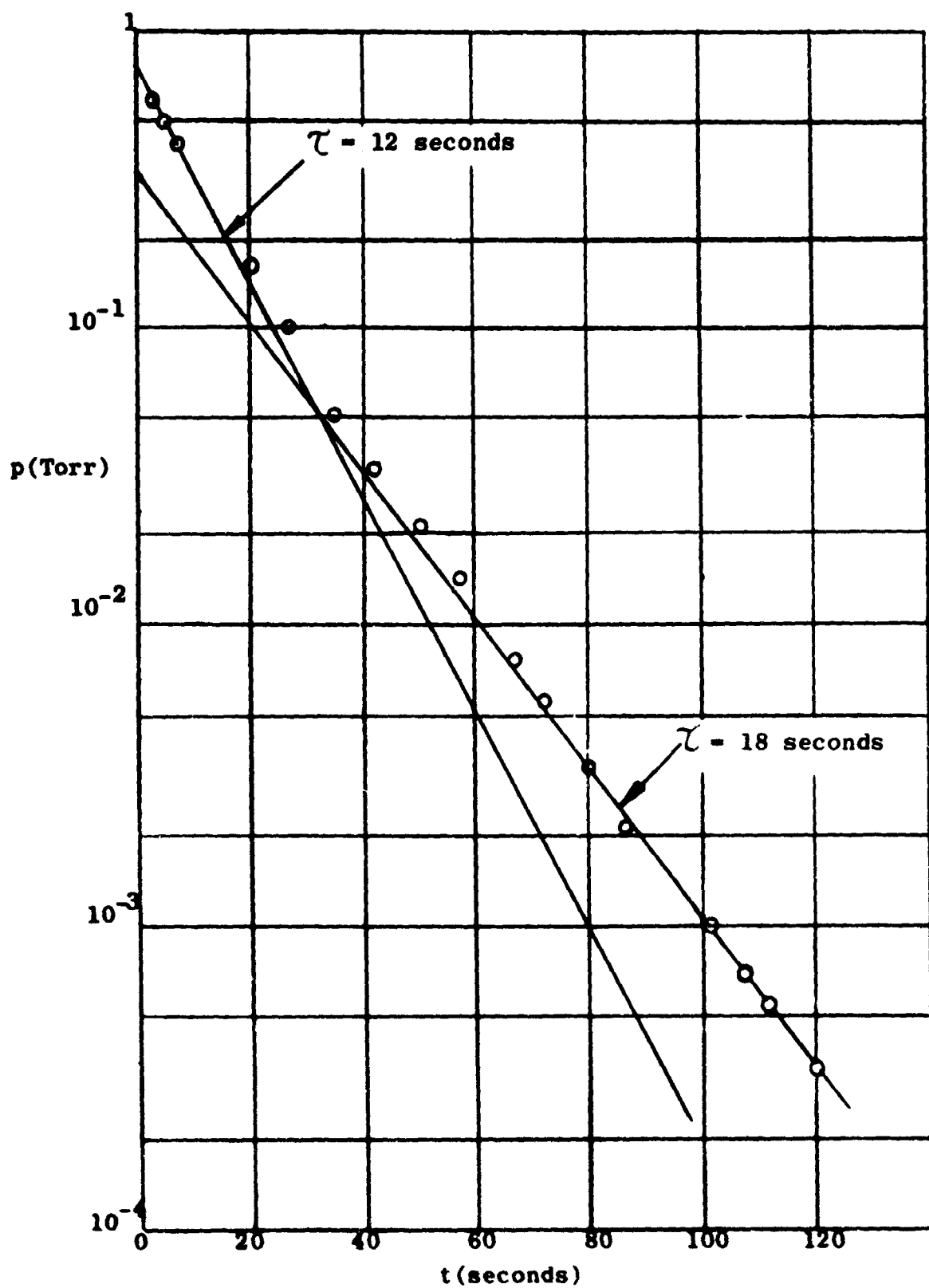
Cryosorption pump chamber, top view.

SECTION III

TEST PROCEDURE

After assembling the system, the entire cryosorption pump section was baked under vacuum, the pump chamber (J) being brought to a final temperature of about 250°C. Several days of baking at this temperature were required to reduce the chamber pressure to a steady value, generally about 2×10^{-6} Torr. The system was then permitted to cool, and an open dewar flask was placed around the pump chamber. After filling the flask with liquid nitrogen, the pump chamber pressure fell rapidly to a value of about 1×10^{-8} Torr. The pressure regulation section was not baked; the pressure in this section was about 5×10^{-6} Torr during bakeout of the cryosorption pump section, and fell below 1×10^{-6} Torr after cooling the cryosorption pump section.

Testing was begun after cooling the pump chamber for several hours in liquid nitrogen. Gas was admitted at atmospheric pressure to the test charge chamber (A), which has about one-thousandth of the combined volume of the expansion chamber (C) and its attached tubing.

The by-pass values (F) and (O) were then closed, and the toggle valve (B) was opened, admitting gas to the expansion chamber. The pressure in this chamber rose to about .76 Torr, as measured with the Alphatron gauge (E), and then declined as the gas escaped through the orifice (D). The rate of decline of pressure should be well approximated by the equation $\dot{p} = Sp/v$, where S is the conductance of the orifice (D), and V is the combined volume of the expansion chamber (C) and its attached tubing. (The conductance of the orifice (K) is entirely negligible compared with that of orifice (D).) Figure 6 shows the observed pressure in the expansion chamber plotted against time, the test gas being air. It is seen that at first the pressure decreases exponentially, with a characteristic time $p/(dp/dt)$ of about 12 seconds. At pressures below 10^{-1} Torr, the rate of decrease slackens, the characteristic time increasing to a value of about 18 seconds at 10^{-4} Torr. This behavior presumably reflects the decrease in the conductance S of the orifice (D) as the flow through the orifice changes from viscous flow ($S \approx 21\ell/$ sec-cm^2) to molecular flow ($S \approx 12\ell/$sec-cm^2).

Pressure in expansion chamber during test.

Figure 6

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The pressure in the cryosorption pump chamber was continuously monitored throughout the burst in the expansion chamber. Figure 7 shows the total pressures, measured with the ionization gauge (L), for air, nitrogen, and argon. The significance of these data will be discussed in the next section. It is worth noting here, however, that the cryosorption pump is less effective for air than for nitrogen. Some evidence supporting this supposition has been obtained with the G. E. 514 partial pressure analyzer whose ionization tube (M) and electron multiplier (N) are shown in Figure 3. Mass spectra were obtained with this instrument by scanning the range between mass 28 and mass 40 at roughly 30 second intervals, after admitting a test charge of air to the system. Figure 8 shows the raw results of this test; it can be seen that the relative concentrations of both oxygen and argon with respect to nitrogen increase in time, indicating a slower pumping speed for the former gases. The instrument is, however, not intended for quantitative analysis. In particular, the gain is non-linear, as evidenced by its markedly higher sensitivity to nitrogen than to oxygen; the ratio of the peak heights for these gases should be roughly 4:1 at the outset of the pressure burst, rather than the observed value of 8:1.
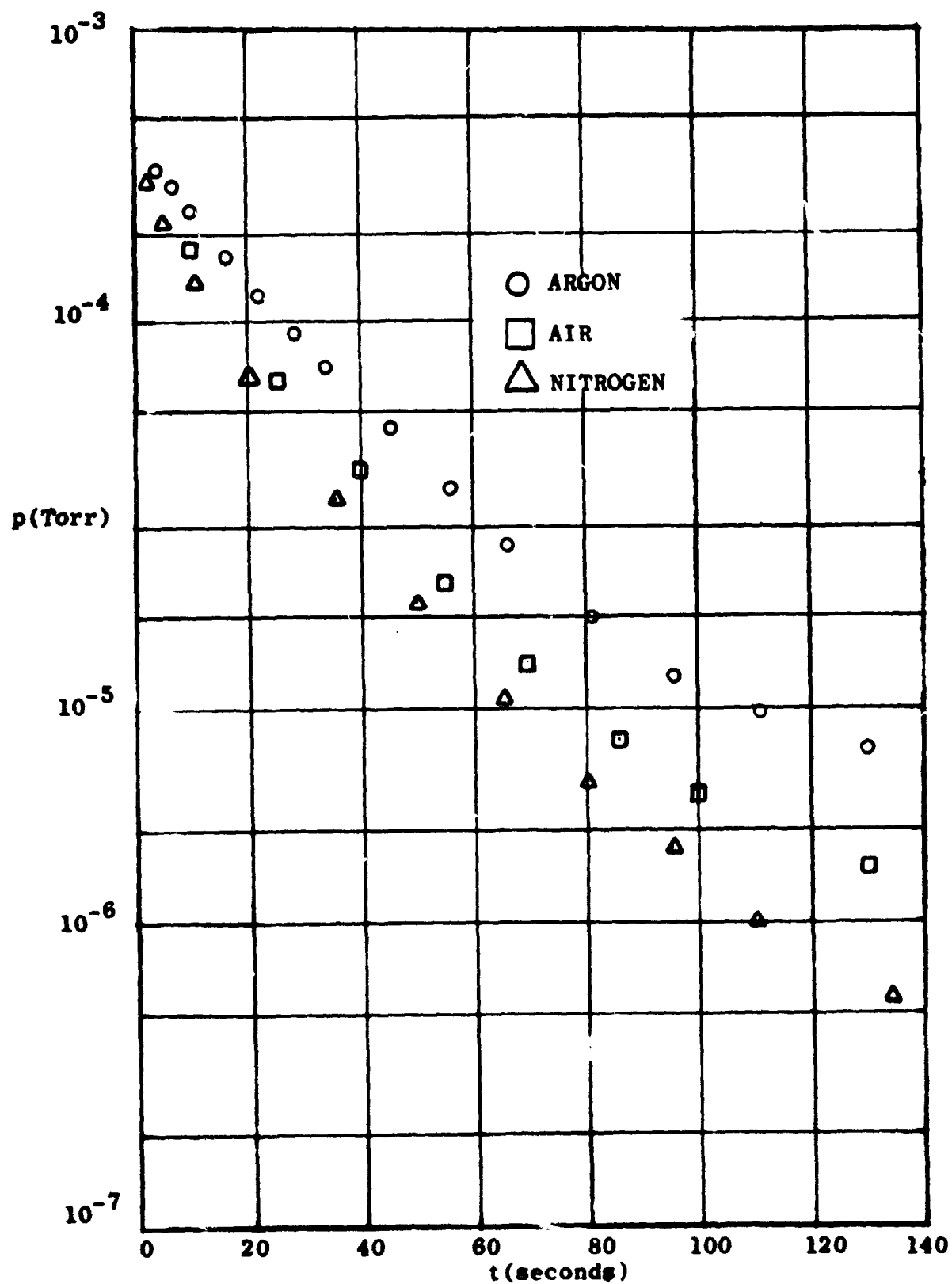
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Figure 7
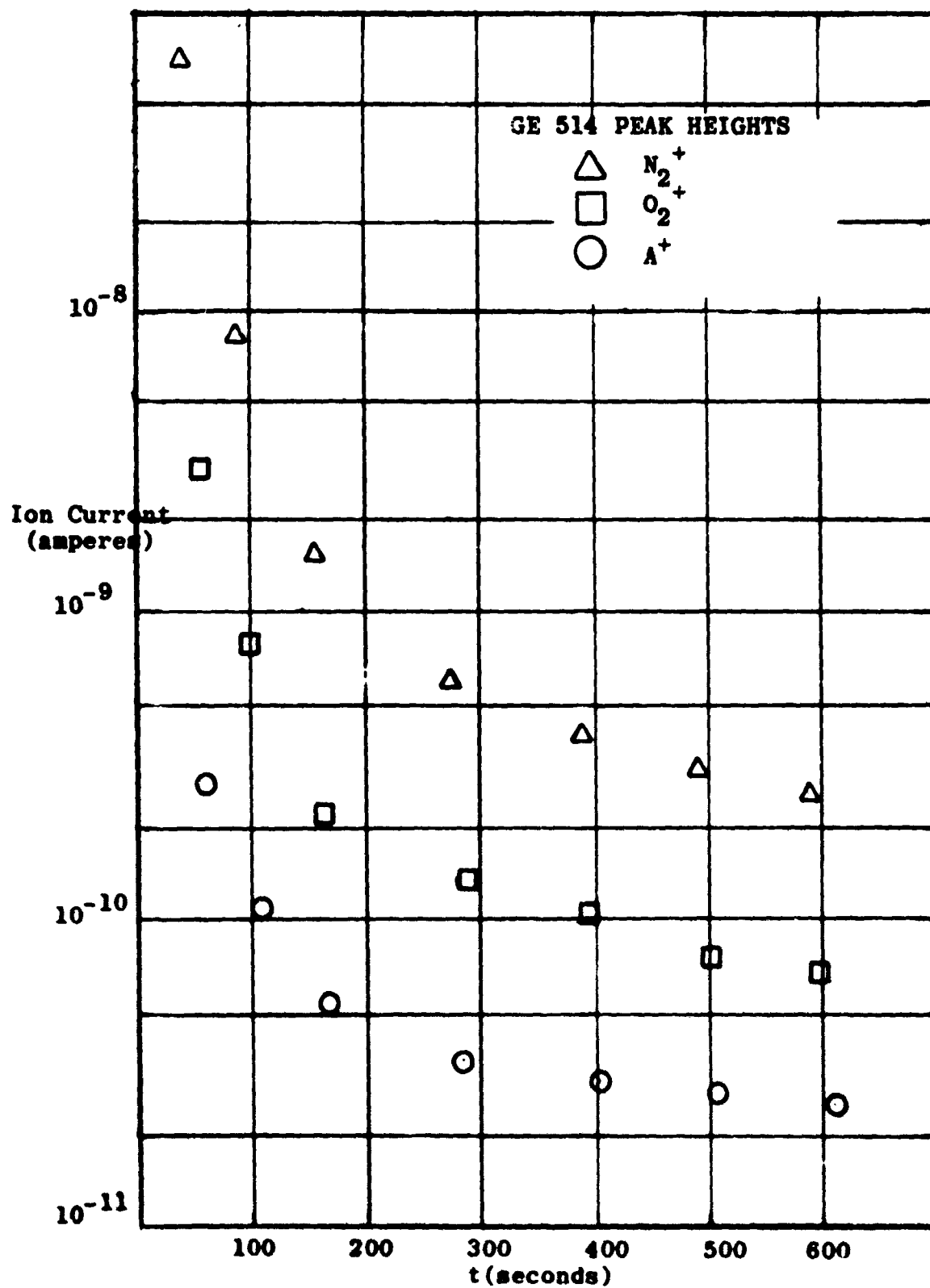Pressure in cryosorption pump chamber during test.

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Figure 8

Relative partial pressures of nitrogen, oxygen, and argon during test.

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SECTION IV

ANALYSIS OF RESULTS

The data presented in Figure 7 can be simply interpreted in terms of the response of a series of ideal impedances and volumes. Let P_{ext} denote the pressure in the regulating section, P_g denote the pressure at the ionization gauge, and P_{cr} denote the pressure at the surface of the cryosorbent. Let R denote the impedance of the orifice connecting the pressure regulating section with the cryosorbent pump section, r denote the impedance between the orifice and the pump (principally that of the radiation shield), and let C denote the volume of the chamber separating R and r. The equation governing the evolution of the measured pressure P_g is then

$$\dot{P_g} = (P_{ext} - P_g)/RC + (P_{cr} - P_g)/rC \qquad (1)$$

Now $P_{ext} \approx P_0 \exp(-t/\tau)$, where $P_0 \approx .76$ Torr and $\tau \approx 12$ sec, $R \approx 100$ sec/ℓ, $C \approx 5\ell$, and $r \ll 1\ell$/sec. Since $rC \ll \tau \ll RC$, the solution to Eq. (1) for all time greater than about 1 second will be

$$P_g \approx P_{cr} + \frac{r}{R} P_{ext} \qquad (2)$$

Thus, the pressure P_{cr} at the surface of the pump can be inferred from the data shown in Figure 7 by subtracting the term $\frac{r}{R} P_{ext}$. Now $\frac{r}{R}$ is readily found by inspection to be about 5×10^{-4}, since $P_{cr} \ll P_g \approx \frac{r}{R} P_{ext}$ in the interval between about 1 second and 10 seconds. Figure 9 shows the pressures P_{cr} estimated in this way for air, nitrogen, and argon. These are the pressures to be expected in the intended flight housing, wherein gas will be admitted to the pump directly from the orifice, rather than through an intervening impedance r.

Finally, we should estimate what fraction of the gas sampled by the intended flight spectrometer will be a residue from an earlier portion of the flight, rather than gas freshly admitted by the orifice. To make this estimate, it is necessary to know the effective ratio of the area of the ionizing region to that of the orifice. It would appear from inspection of a drawing of the
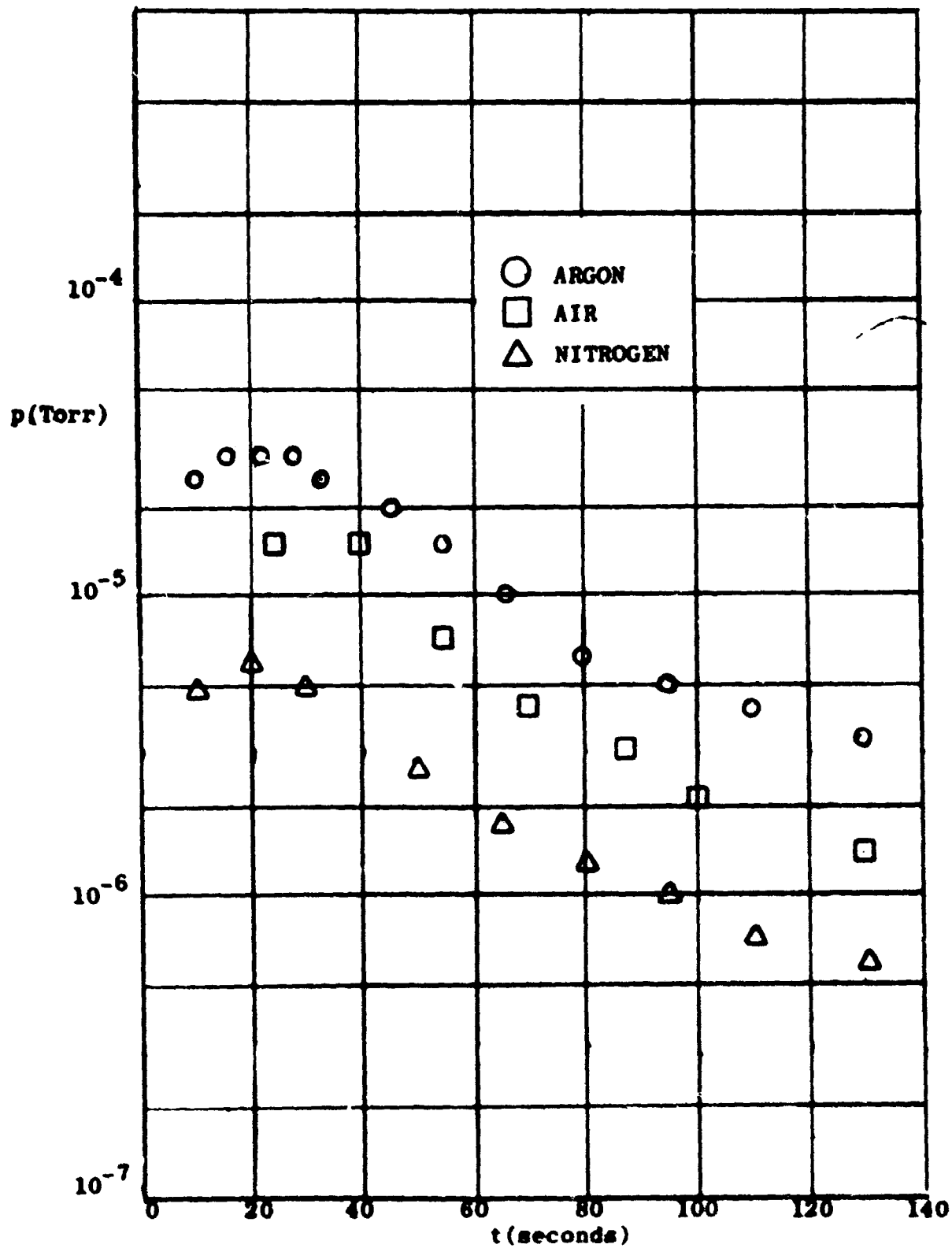
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Figure 9

Estimated pressure at cryosorbent surface during test.

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ion source that this ratio is about 30. Thus, the density of freshly admitt gas within the ionizing region will be one thirtieth of tl . immediately outside the orifice. At an altitude of about 100 K m, the external pressure and temperature are roughly 10^{-4} Torr and $300°K$ respectively, and the density of freshly admitted gas within the ion source will then be about 10^{11} molecules/cc. The residual pressures in the pump will be about 1.5×10^{-6} for nitrogen and about 5×10^{-8} for argon, corresponding at $77°K$ to densities of about 2×10^{11} molecules/cc of residual argon. Thus, at this altitude the residual partial densities would be somewhat larger than those of freshly admitted gas; at lower altitudes, the gas sampled by the spectrometer would be truly representative of the local atmosphere.

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